IS 11321 (1985): Graphite for graphite crucibles [MTD 13: Ores and Raw Materials]

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IS : 11321 - 1985
(Reaffirmed 2003)

Indian Standard

SPECIFICATION FOR GRAPHITE FOR GRAPHITE CRUCIBLES

UDC 666·762·814/·816 : 542·232·54

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3

December 1985

Indian Standard

SPECIFICATION FOR GRAPHITE FOR GRAPHITE CRUCIBLES

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Indian Standard

SPECIFICATION FOR GRAPHITE FOR GRAPHITE CRUCIBLES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 May 1985, after the draft finalized by the Ores and Raw Materials Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Graphite, being an inert material, is most suited for making crucibles for melting ferrous and non-ferrous metals in the industry. Graphite crucibles are required to have high refractoriness, high cold crushing strength and good thermal conductivity. The quality of graphite has vital role for the good thermal efficiency as well as consistency on the service life of graphite crucibles.

0.3 Keeping in view of this, the standardization of crucible grade graphite, was taken up by the relevant technical committee to help in obtaining the right quality of graphite, which will be found useful for the improvement on the quality of the crucibles. The requirements of clay used for the manufacture of clay bonded graphite crucibles are laid down in IS : 4585-1968*.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard covers the requirements of crucible grade graphite for the manufacture of graphite crucibles.

2. SUPPLY OF MATERIAL

2.1 General requirements relating to the supply of graphite for the manufacture of graphite crucibles, shall be laid down in IS : 1387-1967‡.

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*Code of practice for ball-clay for ceramic industry (with Amendment No. 1).
†Rules for rounding off numerical values (*revised*).
‡General requirements for the supply of metallurgical materials (*first revision*).

3. MANUFACTURE

3.1 The material shall be manufactured from crystalline quality flake graphite, silvery in appearance, in the form of fine powder and free from gritty particles.

4. REQUIREMENTS

4.1 The material shall comply with the following chemical and physical requirements.

4.2 Chemical Requirements — All crystalline graphite flakes or fines shall comply with the requirements, on a moisture free basis, as given in Table 1, when tested in accordance with the methods given in Appendix A. Reference to the relevant clauses of Appendix A is also given in Table 1.

TABLE 1 CHEMICAL RFQUIREMENTS OF GRAPHITE FOR GRAPHITE CRUCIBLE

Sl No.	Characteristic	Requirement, Percent by Mass	Method of Test (Ref to Clause No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Moisture	1·0	A-2
ii)	Analysis on dry basis		
	a) Volatile matter, *Max*	1·5	A-3
	b) Ash:	—	A-4
	Fe_2O_3, *Max*	1·2	A-4.2
	TiO_2, *Max*	0·5	
	CaO, *Max*	0·02	
	$SiO_2 + Al_2O_3$, *Max*	Balance	
	Alkalis, *Max*	0·2	
	c) Fixed carbon, *Min*	85·0	A-5
iii)	Sulphur, *Max*	0·25	A-6

4.3 Physical Requirements — All crystalline graphite shall be free from oil, uniform in quality, free from visible impurities, such as mica, pyrites and free from other contaminations.

4.3.1 *Fineness* — The fineness of graphite shall confirm to the requirement specified in Table 2.

4.3.2 *Flakiness Index* — The flakiness index which a reciprocal measure of packing density, when determined by the method given in Appendix **A-7** shall not be less than 125.

TABLE 2 MINIMUM PERCENTAGE OF MATERIAL SIZE RANGE

(*Clause* 4.3.1)

Sieve No.*	− 1·20 mm + 180 μm	− 300 μm	− 1·20 mm + 125 μm	− 1·20 mm + 300 μm
Percent	100	< 5	75	95

**See* IS : 460 (Part 1)-1978 Wire cloth test sieves (*second revision*).

4.3.3 *Oxidation Temperature* — The oxidation temperature of graphite flakes shall be approximately 750°C. The method for determination of oxidation temperature may be by mutual agreement.

5. SAMPLING

5.1 Representing samples of the material shall be drawn as prescribed in Appendix B.

6. PACKING

6.1 Unless specified otherwise, the material shall be supplied in moisture proove bags, containers or packets, each containing 25 kg.

7. MARKING

7.1 Each bag, container or packet shall be clearly marked with the manufacturer's name or trade-mark and the grade of the material.

7.2 The material may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

APPENDIX A

(*Clauses* 4.3, 4.3.1 and *Table* 1)

METHODS OF TEST

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals shall be employed in the tests and distilled water (*see* IS : 1070-1977*) shall be used where the use of water as reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

A-2. DETERMINATION OF MOISTURE

A-2.1 Procedure — Weigh accurately about 5 g of the material in a tared porcelain dish and cover it with a watch-glass. Place the dish in an air-oven maintained at 150 ± 1°C and remove the watch-glass from it. Heat the sample for two hours in the oven and cover the dish with the watch-glass before taking it out of the oven. Cool the dish in a desiccator and weigh. Repeat heating, cooling and weighing to constant weight.

A-2.2 Calculation

$$\text{Moisture, percent} = \frac{A}{B} \times 100$$

where

A = loss in weight in g of the material after heating, and

B = weight in g of the material taken.

A-3. DETERMINATION OF VOLATILE MATTER

A-3.1 Procedure — Introduce in a weighed platinum crucible with tightly fitting lid about one gram of the moisture-free sample. The lid should have a hole, one millimetre in diameter, in the middle to help the escape of volatile matter. Heat the crucible in a muffle furnace maintained at a temperature of 925 ± 25°C for 7 minutes. The bottom of the crucible shall not rest on the muffle floor. Remove the crucible from the muffle after 7 minutes; cool in a desiccator and weigh.

*Specification for water for general laboratory use (*second revision*).

A-3.2 Calculation

$$\text{Volatile matter, percent} = \frac{A}{B} \times 100$$

where

A = loss in weight in g of the moisture-free sample after heating up to 925 ± 25°C, and

B = weight in g of the moisture-free sample taken.

A-4. DETERMINATION OF ASH

A-4.1 Procedure — Weigh accurately about 10 to 20 g of the moisture free material in a tared platinum dish, keep in a muffle furnace and heat to 500 ± 10°C within one hour and 775 ± 10°C in two hours. A slow stream of air is maintained through the muffle furnace. When the carbon is completely removed as indicated by the absence of black particle upon stirring with a platinum wire, the temperature is further increased to 950 ± 25°C and kept for one hour. Cool in a desiccator and weigh. Repeat this procedure till the residue in the crucible is constant in weight.

A-4.1.1 *Calculation*

$$\text{Ash, percent} = \frac{A}{B} \times 100$$

where

A = weight of ash in g, and

B = weight of the sample taken.

A-4.2 Determination of Fe_2O_3, TiO_2, CaO, SiO_2, Al_2O_3 and alkalis (Na_2O and K_2O).

A-4.2.1 *Procedure* — The ash taken from **A-3.1** is fused with a fusion mixture and the chemical analysis of ash shall be determined in accordance with the procedure specified in IS : 1527-1972*.

A-5. DETERMINATION OF FIXED CARBON

A-5.1 Procedure — The fixed carbon content of the graphite is determined as difference of the total graphite (moisture free) taken and sulphur, volatile matter and ash (all in percentage basis) and reported as fixed carbon of the graphite.

*Methods for chemical analysis of high silica refractory materials (*first revision*).

A-6. DETERMINATION OF SULPHUR

A-6.1 Reagents

A-6.1.1 *Barium Chloride Solution (100 g/litre)* — Dissolve 100 g of barium chloride ($BaCl_2 \cdot 2H_2O$) and dilute to 1 litre with water.

A-6.1.2 *Bromine Water (Saturated)* — Add an excess of bromine to 1 litre of water.

A-6.1.3 *Eschka Mixture* — Thoroughly mix 2 parts by weight of light calcined magnesium oxide (MgO) with 1 part of anhydrous sodium carbonate (Na_2CO_3). Both materials should be as free as possible from sulphur.

A-6.1.4 *Concentrated Hydrochloric Acid — See* IS : 265-1962*.

A-6.1.5 *Dilute Hydrochloric Acid* — Approximately 6N.

A-6.1.6 *Sodium Carbonate, Saturated Solution* — Dissolve approximately 60 g of crystallized sodium carbonate ($Na_2CO_3 \cdot 10H_2O$) or 22 g of anhydrous sodium carbonate (Na_2CO_3) in 100 ml of water, using a sufficient excess of Na_2CO_3 to ensure a saturated solution.

A-6.1.7 *Sodium Hydroxide Solution (100 g/litre)* — Dissolve 100 g of sodium hydroxide (NaOH) in 1 litre of water. This solution may be used in place of the Na_2CO_3 solution.

A-6.2 Procedure — Weigh accurately about 1 g of moisture free graphic powder (250 μm) and mix it with Eschka Mixture. Transfer the mixture to a porcelain crucible and cover with a layer of Eschka Mixture. Place the crucible in a cold muffle furnace and increase the temperature gradually to about 800 $\pm$ 25°C. Maintain the temperature until all the particle disappear. Cool the mixture to room temperature, then transfer it to a beaker and digest with hot water. Filter the solution, add bromine water and acidify with 6N HCl. Boil to remove excess bromine. Adjust the solution to *p*H7 by adding alkali (sodium carbonate or sodium hydroxide) and then add 1 ml of 1N HCl. The sulphur in graphite which has been converted to sodium sulphate is then precipitated by barium chloride solution. Filter the solution after digesting for 2 hours and wash the barium sulphate precipitate. Ignite this precipitate at 925 $\pm$ 25°C till a constant weight is achieved. Calculate the sulphur content of the graphite from the weight of barium sulphate.

*Specification for hydrochloric acid.

A-7. FLAKINESS INDEX

A-7.1 Procedure — Weigh about 100 g of graphite and pour it into a graduated measuring cylinder of 500 ml capacity. After pouring the graphite, tap the cylinder several times to settle the particles at the bottom of the cylinder. Make the top surface flat by placing a flat bottomed cylindrical wooden piece. Measure the volume occupied by the graphite in the cylinder from the graduated mark on the cylinder. Report the flakiness index of the graphite as the volume in millilitre occupied by graphite flake.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF GRAPHITE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being supplied, the sampling instrument, and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight metal or glass containers.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight by suitable means after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In any consignment all the bags/packets of the material of the sample grade and drawn from the same batch of manufacture shall be grouped together to constitute a lot.

B-2.2 Tests for determination of the conformity of a lot to the requirements of the specification shall be done from each lot separately. The number of bags/packets to be selected at random for this purpose from a lot shall be in accordance with Table 3.

TABLE 3 SCALE OF SAMPLING

No. of Bags/Packets in the Lot	No. of Bags/Packets to be Selected
Up to 50	3
51 ,, 100	4
101 ,, 300	5
301 ,, 500	7
501 and above	10

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the bags/packets selected according to **B-2.2** small portions of the material shall be drawn with the help of a suitable sampling insrument from different parts of the bag/packet. The total quantity of the material so drawn from each bag/packet shall be not less than 500 g and these shall form the individual samples representative of the different bags/packets selected.

B-3.2 From each of the individual samples formed according to **B-3.1**, not less than 300 g of material shall be drawn and mixed together to form a composite sample. The composite sample so formed may be reduced further, if necessary, by coning and quartering so as to obtain enough material sufficient to conduct all the tests specified in the standard.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of all the characteristics listed in Table 1 shall be conducted on each of the individual samples (*see* **B-3.1**).

B-4.2 Tests for the determination of fineness of the material as given in Table 2 shall be conducted on the composite sample (*see* **B-3.2**).

B-5. CRITERIA FOR CONFORMITY

B-5.1 The lot shall be declared as conforming to the requirements of the specification if the conditions stipulated in **B-5.1.1** and **B-5.1.2** are specified.

B-5.1.1 From the individual test results, the mean and the range shall be calculated wherever possible. (Range is defined as the difference between the maximum and minimum values of test results.)

B-5.1.2 For each of the characteristics listed in Table 1 the value of the corresponding expression (mean + 0·6 range) shall be found to be less than or equal to the relevant maximum value specified.

B-5.1.3 The test results for fineness of the material determined on the composite sample shall be found to be satisfactory as given in Table 3.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	k
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2